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NEWS	14	MAY 15	INPADOCDB and INPAFAMDB enhanced with Chinese legal status data
NEWS	15	MAY 28	CAS databases on STN enhanced with NANO super role in records back to 1992
NEWS	16	JUN 01	CAS REGISTRY Source of Registration (SR) searching enhanced on STN

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FILE 'HOME' ENTERED AT 15:46:42 ON 03 JUN 2009

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=> file registry
COST IN U.S. DOLLARS                                SINCE FILE      TOTAL
                                                         ENTRY    SESSION
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DICTIONARY FILE UPDATES:   2 JUN 2009  HIGHEST RN 1151889-97-2

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TSCA INFORMATION NOW CURRENT THROUGH January 9, 2009.

  Please note that search-term pricing does apply when
  conducting SmartSELECT searches.

REGISTRY includes numerically searchable data for experimental and
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http://www.cas.org/support/stngen/stndoc/properties.html

=>
Uploading C:\Program Files\Stnexp\Queries\10562142.str

L1      STRUCTURE UPLOADED

=> d l1
L1 HAS NO ANSWERS
L1              STR
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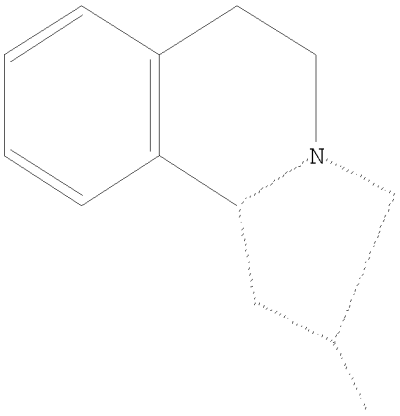

Structure attributes must be viewed using STN Express query preparation.

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=> s l1
SAMPLE SEARCH INITIATED 15:47:23 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED -     4214 TO ITERATE

 47.5% PROCESSED     2000 ITERATIONS                                25 ANSWERS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS:  ONLINE  **COMPLETE**
                        BATCH   **COMPLETE**
PROJECTED ITERATIONS:        80387 TO    88173
PROJECTED ANSWERS:             618 TO     1488

L2           25 SEA SSS SAM L1

=> s l1 ful
FULL SEARCH INITIATED 15:48:02 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED -    87035 TO ITERATE

100.0% PROCESSED    87035 ITERATIONS                              795 ANSWERS
SEARCH TIME: 00.00.01

L3          795 SEA SSS FUL L1

=> file caplus
COST IN U.S. DOLLARS                            SINCE FILE      TOTAL
                                                   ENTRY    SESSION
FULL ESTIMATED COST                               186.36     186.58

FILE 'CAPLUS' ENTERED AT 15:48:05 ON 03 JUN 2009
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FILE COVERS 1907 - 3 Jun 2009  VOL 150 ISS 23
FILE LAST UPDATED: 2 Jun 2009  (20090602/ED)
REVISED CLASS FIELDS (/NCL) LAST RELOADED:  Feb 2009
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE:  Feb 2009

CAplus now includes complete International Patent Classification (IPC)
reclassification data for the third quarter of 2008.
```

CAS Information Use Policies apply and are available at:

http://www.cas.org/legal/infopolicy.html

This file contains CAS Registry Numbers for easy and accurate

=> s l3
L4 169 L3

=> file registry
COST IN U.S. DOLLARS SINCE FILE ENTRY TOTAL SESSION
FULL ESTIMATED COST 2.00 188.58

FILE 'REGISTRY' ENTERED AT 15:50:24 ON 03 JUN 2009
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STRUCTURE FILE UPDATES: 2 JUN 2009 HIGHEST RN 1151889-97-2
DICTIONARY FILE UPDATES: 2 JUN 2009 HIGHEST RN 1151889-97-2

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TSCA INFORMATION NOW CURRENT THROUGH January 9, 2009.

Please note that search-term pricing does apply when
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REGISTRY includes numerically searchable data for experimental and
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http://www.cas.org/support/stngen/stndoc/properties.html

=>
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L5 STRUCTURE UPLOADED

=> s l5 ful
FULL SEARCH INITIATED 15:50:43 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 46310 TO ITERATE

100.0% PROCESSED 46310 ITERATIONS 515 ANSWERS
SEARCH TIME: 00.00.01

L6 515 SEA SSS FUL L5

=> file caplus
COST IN U.S. DOLLARS SINCE FILE ENTRY TOTAL SESSION

FULL ESTIMATED COST 185.88 374.46

FILE 'CAPLUS' ENTERED AT 15:50:53 ON 03 JUN 2009

FILE COVERS 1907 - 3 Jun 2009 VOL 150 ISS 23
FILE LAST UPDATED: 2 Jun 2009 (20090602/ED)
REVISED CLASS FIELDS (/NCL) LAST RELOADED: Feb 2009
USPTO MANUAL OF CLASSIFICATIONS THESAURUS ISSUE DATE: Feb 2009

CAplus now includes complete International Patent Classification (IPC) reclassification data for the third quarter of 2008.

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This file contains CAS Registry Numbers for easy and accurate

=> s l6
L7 91 L6

=> d abs bib fhitstr 80-91

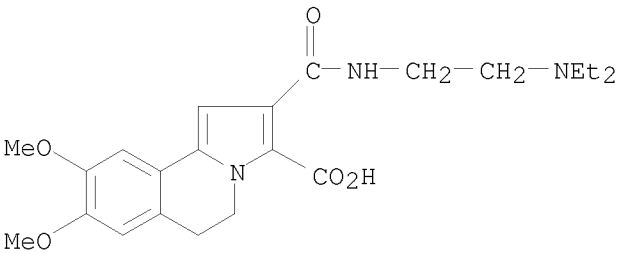
L7 ANSWER 80 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB The title compds. (I) (R = H, CO2H, or CO2R; R1 = H, CO2H, or CH2CO2H acids, esters, or amides, or alkyl, cycloalkyl or aryl; and R2 = H, CO2H, or CO2R), are hypotensive, sympathicolytic, and psychotropic agents. They are synthesized by the reaction of a 6,7-dimethoxy-3,4-dihydroisoquinoline (II) with a 2-Cl or [-Br ketone. Thus, a mixture of 50 g. 1-Me derivative of II, 39 g. Et chloropyruvate, and 42 g. NaHCO3 in 500 ml. of EtOH was stirred at 35° 5 hrs., and the mixture diluted with 1.5 l. H2O, filtered], and washed with H2O to give I (R = R1 = H, R2 = CO2Et) (III), m. 111-13° (EtOH-ligroine). Other I similarly prepared were: (R, R1, R2, and m.p. given): H, CH2CO2Et H, (IV), 91-3°; CO2Et, Ph, H (V), 172-4°; H, CO2Et, CO2Et (VI), 91-3°; H, Ph, H (VII), 136-40°; H, cyclohexyl, H (VIII), 122-4°. A solution of 10 ml. 13% NaOEt in EtOH was added to a solution of 48 g. V and 20 g. Me2NC2H4OH in 600 ml. PhMe, the mixture refluxed 6 hrs. (removing EtOH as an azeotrope), cooled, washed with H2O, and extracted with HOAc, the extract made alkaline with NH3
and extracted with CHCl3, and the CHCl3 evaporated to give I (R = CO2C2H4NMe2, R1

= Ph, R2 = H), m. 137-9° (EtOH). Similarly prepared I were (R, R1, R2, and m.p. given); H, Me, CO2C2H4NMe2, 98-9° (HCl salt m. 252-5°); H, Me, CO2(CH2)3Q (Q = piperidino), 102-4°; H, H, CONHC2H4NEt2, 146-8°. Hydrolysis of the Et esters with boiling alc. NaOH gave the corresponding acids (I ester hydrolyzed and m.p. of acid given): III, 232-4° (decomposition); IV, 159-60°; V, 219-11° (decomposition); VI, 229-30° (anhydride IX m. 239-40°). Treatment of IX with Et2NC2H4NH2 gave I (R = H, R1 = Et2NC2H4NHCO, and R2 = CO2H), m. 168-70°. A solution of 20 g. VII in 1.6 ml. HOAc was reduced with 3-20 atmospheric of H using 3 g. Pt oxide 25-30 hrs. at ambient temperature to give 2-phenyl-1,2,3,5,6,10b-hexahydro-8,9-dimethoxypyrrolo[2,1-a]isoquinoline, m. 121-3°. Reduction of VII with Raney Ni at 100° in EtOH at 130 atmospheric gave a mixture of VIII and I (R = H, R1 = cyclohexyl, R2 = H), m. 122-4°, and 2-cyclohexyl-1,2,3,5,6,10b-hexahydro-8,9-dimethoxypyrrolo-[2,1-a]isoquinoline, m. 91-2°; sulfate m. 170-1°; HBr3 salt m. 146-8°.

AN 1969:481215 CAPLUS
DN 71:81215
OREF 71:15049a,15052a
TI Hypotensive pyrrolo [2,1-a] isoquinoline
IN Ferrari, Giorgio; Casagrande, Cesare
PA SIPHAR S. A.
SO Brit., 8 pp.
CODEN: BRXXAA
DT Patent
LA English
FAN.CNT 1

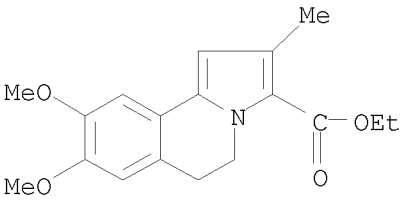
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI GB 1153670		19690529	GB 1967-55371	19671205
FR 1555788			FR	
FR 7348			FR	
PRAI BE		19661207		

IT 17606-23-4P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 17606-23-4 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-3-carboxylic acid, 2-[[[2-(diethylamino)ethyl]amino]carbonyl]-5,6-dihydro-8,9-dimethoxy- (CA INDEX NAME)



L7 ANSWER 81 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN

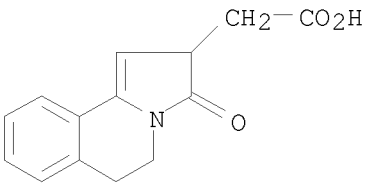
AB A series of compds. with the pyrrolo[2,1-a]isoquinoline ring system was synthesized by Tschitschibabin cyclization and subsequent transformations. The pharmacol. activity of the new compds., was studied.
AN 1968:427223 CAPLUS
DN 69:27223
OREF 69:5063a,5066a
TI Synthesis and pharmacological evaluation of some pyrrolo[2,1-α]isoquinolines
AU Casagrande, Cesare; Invernizzi, Ambrogio; Ferrini, Rosano; Ferrari, Giorgio G.
CS Res. Lab., Simes S.p.A., Milan, Italy
SO Journal of Medicinal Chemistry (1968), 11, 765-70
CODEN: JMCMAR; ISSN: 0022-2623
DT Journal
LA English
OS CASREACT 69:27223
IT 2683-23-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 2683-23-0 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-3-carboxylic acid, 5,6-dihydro-8,9-dimethoxy-2-methyl-, ethyl ester (CA INDEX NAME)



L7 ANSWER 82 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Reaction of 1-(R-substituted)-3,4-dihydroisoquinolines (I) with maleic, bromomaleic, and citraconic anhydrides (II), (III), and (IV), resp., and with fumaric, bromomaleic, and citraconic acids (V), (VI), and (VII), resp., gave the title pyrrocoline derivs. (VIII). Thus, 0.01 mole II was slowly added to a solution of 0.01 mole I in 10 cc. C6H6 and after 4 hrs. VIIIa-f were isolated. A solution of 0.01 mole III in 5 cc. C6H6 slowly added to a cooled solution of 0.01 mole I in 10 cc. C6H6 gave VIIIg-j. III (0.01 mole) was slowly added to 0.01 mole I and the mixture kept 24 hrs. to give VIIIk-p. The following VIII were obtained [compound, R1, R2, m.p., and % yield given]: a, H, H, 190-5°, 37.1; b, Ph, H, 90-2°, 92.5; c, CO2Me, H, 133-7° (C6H6), 96.0; d, CO2Et, H, 113-14° (hexane), 95.5; e, (CH2)2CN, H, 95-8° (hexane), 91.7; f, (CH2)2CO2Me, H, 95° (hexane), 92.0; g, H, Br, 110-15° (Me2CO-Et2O), 40.9; h, Ph, Br, 110° (Et2O), 36.7; i, (CH2)2CN, Br, 90-5° (Me2COEt2O), 32.9; j, (CH2)2CO2Me, Br, 120-5° (AcOH-H2O), 31.8; k, H, Me, 75-8° (C6H6-hexane), 15.5; l, Ph, Me, 75-80°, 36.3; m, CO2Me, Me, 105-10°, 22.0; n, CO2Et, Me, 110° (50% AcOH), 71.2; o, (CH2)2CN, Me, 75-80° (C6H6-hexane), 71.2; p, (CH2)2CO2Me, Me, 75-80° (Et2O), 11.8. A mixture of 1.25 g. I (R = Me) and 5.8 g. V was heated at 150° 2 hrs.,

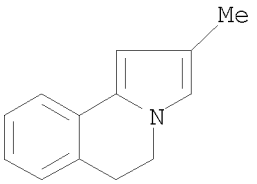
dissolved in 10% NaOH, extracted several times with Et2O, filtered, and acidified with HCl to obtain 2 g. VIIIa. A mixture of 1.87 g. 1-methylene-N-acetyl-1,2,3,4-tetrahydroisoquinoline and 1.74 g. V was heated at 130-50° 2.5 hrs. and the mixture worked up as above to give 0.7 g. VIIIa. Similarly, VIIIb was prepared from equimolar amts. of I (R = PhCH2) and V, and from V and 1-benzylidene-N-acetyl-1,2,3,4-tetrahydroisoquinoline. A mixture of 2.21 g. I (R = PhCH2) and 1.3 g. VII was heated at 130° 3 hrs. and the product dissolved in hot 10% NaOH and precipitated with HCl to give 2.25 g. VIIIb, m. 95-100°. A mixture of 2.2 g. I (R = PhCH2) and 1.95 g. VI was heated at 110-20° 30 min., dissolved in EtOH, and added slowly to Et2O to give 0.65 g. VIIIh, m. 180°. The structure of VIII was confirmed by ir spectra. VIIIj decompose on long standing or upon heating to a compound (IX), m. 165-70° (EtOH-H2O), which does not contain Br. The structure of IX was suggested on the basis of its ir spectrum.

AN 1968:114409 CAPLUS
DN 68:114409
OREF 68:22046h,22047a
TI Reactivity of the methyl group in 1-methyl-3,4-dihydroisoquinoline. IV. Synthesis of mono- and dicarboxylic acids of the 5,6-dihydrobenzo[g]pyrrocoline series
AU Agbalyan, S. G.; Nersesyan, L. A.; Nshanyan, A. O.
SO Armyanskii Khimicheskii Zhurnal (1967), 20(6), 447-53
CODEN: AYKZAN; ISSN: 0515-9628
DT Journal
LA Russian
IT 18121-49-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 18121-49-8 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-2-acetic acid, 2,3,5,6-tetrahydro-3-oxo- (CA INDEX NAME)

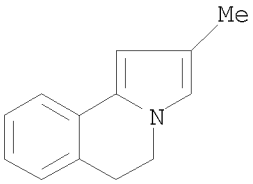


L7 ANSWER 83 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
AB cf. CA 65, 8979b. Aryl-1-isoquinolylmethyl benzoates prepared from 2-benzoyl-1,2- dihydroisoquinaldonitrile were hydrolyzed to aryl-1-isoquinolylmethanols. These alcs. were oxidized to the corresponding ketones and reduced to the corresponding 1-benzylisoquinolines.
AN 1966:499250 CAPLUS
DN 65:99250
OREF 65:18559a-b
TI Reissert compound studies. XIII. Model reactions based on 2-benzoyl-1,2-dihydroisoquinaldonitrile
AU Gibson, H. W.; Popp, F. D.

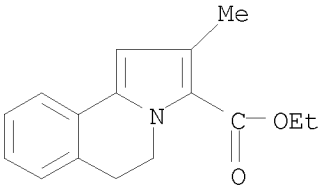
CS Clarkson Coll. of Technol., Potsdam, NY
SO Journal of the Chemical Society [Section] C: Organic (1966), (20), 1860-4
CODEN: JSOOAX; ISSN: 0022-4952
DT Journal
LA English
IT 10174-46-6
(Derived from data in the 7th Collective Formula Index (1962-1966))
RN 10174-46-6 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline, 5,6-dihydro-2-methyl- (CA INDEX NAME)



L7 ANSWER 84 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Preparation of I was reported. Thus, into an ice-cooled solution of 3 g. 1-methyl-3,4-dihydroisoquinoline in 10 ml. C6H6 is added 2.8 g. bromoacetone, the mixture kept in a refrigerator overnight, C6H6 is removed, the residue washed with Et2O, warmed 5 hrs. at 50° with 40 ml. 5% Na2CO3 solution, and extracted with Et2O to give 352 mg. I (R1 = Me, R2 = H), m. 23-5°. Similarly prepared are the following I (R1, R2, m.p., and % yield given): Ph, H, 114° 36; Ph, OMe, 138° 47; (CH2)2CO2Me, H, 80° 30; (CH2)2CO2Me, OMe, 109° 68. Also prepared are 5,6-dihydropyrrolo[2,1-a]-β-carboline, m. 196°, and 2-methyl-3-ethoxycarbonyl-5,6-dihydropyrrolo[2, 1-a]-β-carboline, m. 244° (decomposition).
AN 1966:499249 CAPLUS
DN 65:99249
OREF 65:18558g-h,18559a
TI Synthesis of 5,6-dihydropyrrolo[2,1-a]isoquinolines
AU Sakai, Shinichiro; Kubo, Akinori; Inaba, Minoru; Katagiri, Michiko; Tanno, Kayoko
CS Univ. Chiba, Japan
SO Yakugaku Zasshi (1966), 86(9), 856-8
CODEN: YKKZAJ; ISSN: 0031-6903
DT Journal
LA Japanese
IT 10174-46-6P, Pyrrolo[2,1-a]isoquinoline, 5,6-dihydro-2-methyl-
RL: PREP (Preparation)
(preparation of)
RN 10174-46-6 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline, 5,6-dihydro-2-methyl- (CA INDEX NAME)



L7 ANSWER 85 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB A study of the reaction of chloral and Et diazoacetate as a potential source of Et trichloroacetoacetate (I) showed that the main product of this reaction was Et 3-(trichloromethyl)glycidate. The reaction of trichloroacetyl chloride, ketene, and an alc., in liquid SO2, was found to be an excellent method to prepare trichloro-β-oxo esters. The acid hydrolysis of I yielded α,α,α-trichloroacetone but this reaction could not be utilized as a general synthetic route to trichloromethyl ketones because alkylation of the ester could not be accomplished. The reactions of I with amines were studied and the products formed depended on the basicity and structure of the amine. NH3 reacted with the ester to form Et malonamate. Primary aliphatic amines yielded malonamides and secondary amines formed amine salts. Aromatic amines did not react with I under similar conditions but in the presence of polyphosphoric acid they gave 2-trichloromethyl-4-quinolones. These compds. could be hydrolyzed to kynurenic acids (II), thus providing a new synthetic route to these compds. The condensation of I with o-phenylenediamine, under neutral conditions, yielded 4-(trichloromethyl)-1H-1,5-benzodiazepin-2(3H)-one. 32 references.
AN 1966:499248 CAPLUS
DN 65:99248
OREF 65:18558e-g
TI Trichloroacetoacetates. I. Synthesis and reactions of ethyl and β,β,β,-trifluoroethyl trichloroacetoacetates
AU Wald, David K.; Joullie, Madeleine M.
CS Univ. of Pennsylvania, Philadelphia
SO Journal of Organic Chemistry (1966), 31(10), 3369-74
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA English
OS CASREACT 65:99248
IT 10174-78-4P, Pyrrolo[2,1-a]isoquinoline-3-carboxylic acid, 5,6-dihydro-2-methyl-, ethyl ester
RL: PREP (Preparation)
(preparation of)
RN 10174-78-4 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-3-carboxylic acid, 5,6-dihydro-2-methyl-, ethyl ester (CA INDEX NAME)

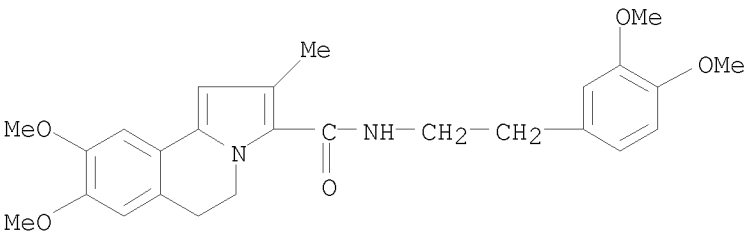


L7 ANSWER 86 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB cf. CA 56, 14343f. The synthesis of C-bisnorrubremetinium salts (I) and related compds. without an oxidation step confirmed formula II for the rubremetinium cation. N.M.R. data confirmed this structure. Ir and uv data are also given. Refluxing an absolute EtOH solution containing 4 g. 1-methyl-6,7-dimethoxy-3,4-dihydroisoquinoline and 1.6 g. Et chloroacetate 2 hrs. gave, after standing overnight, 2.9 g. Et 2-methyl-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-3-carboxylate (III), m. 91-2°. III was converted to hydrazide on heating with 100% NH2NH2 10 hrs. (oil bath at 150°), m. 197-9°. The hydrazide (0.5 g.) in dilute AcOH was converted to azide with NaNO2 in aqueous solution and worked up in ether solution To the dried solution was added 0.5 g. 3,4-dimethoxyphenethylamine in 20 cc. dry ether with cooling. The mixture was evaporated, benzene added, and the solution refluxed 3 hrs. to give 0.22 g. 2-methyl-3-[3-(3,4-dimethoxyphenethyl)ureido]-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline, m. 195-7'. III (1 g.) was hydrolyzed in 10% alc. KOH. The K salt, obtained in 1.1-g. yield, was dissolved in 20 cc. H2O, and 0.18 g. AcOH in 5 cc. H2O was added with cooling. On standing overnight 0.87 g. 2-methyl-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-3-carboxylic acid (IV), m. 160-2° (decomposition), was obtained. Refluxing a solution of 40 mg. IV in 10 cc. benzene and recrystg. the product from hexane gave 35.5 mg. 2-methyl-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline, m. 110-10.5°. To a solution of 1.64 g. IV and 1.05 g. 3,4-dimethoxyphenethylamine in 30 cc. CHCl3 was added 3 g. dicyclohexylcarbodiimide. After 3 days the precipitated urea was filtered off and the excess reagent was decomposed with 5% AcOH. The filtered, washed, and dried (K2CO3) CHCl3 layer was evaporated to give 0.60 g. N-(3,4-dimethoxyphenethyl)-2-methyl-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-3-carboxamide (V), m. 171-3°. To a refluxing solution of 200 mg. V in 20 cc. dry toluene was added 2 g. P2O5 in 3 portions at 0.5-hr. intervals; the mixture was refluxed 2 hrs. to give 89 mg. 2-methyl-3-(6,7-dimethoxy-3,4-dihydro-1-isoquinolyl)-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline (VI), m. 165-7° (petr. ether). VI (100 mg.) on refluxing (steam bath) 2 hrs. in benzene with 29 mg. Me2SO4 gave 127 mg. quaternary methosulfate (VII), red prisms, m. 176-7° (EtOH-benzene). To a solution of 27.8 g. 1-methyl-6,7-dimethoxy-3,4-dihydroisoquinoline in 150 cc. absolute EtOH was added with cooling a solution of 16.1 g. diethyl 2-chloro-3-oxopentanedioate, b12 146.5-7.5°, in 50 cc. absolute EtOH and the mixture refluxed 30 min. to give 14.2 g. Et 3-ethoxycarbonyl-8,9-dimethoxy- 5,6 - dihydropyrrolo[2,1 - a]isoquinoline-2-acetate (VIII), m. 95-7° (hexane-EtOH). Hydrolysis of VIII with alc. KOH gave 3-carboxy-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-2-acetic

acid (IX), m. 160-1°, in 81% yield. Decarboxylation in refluxing xylene 2 hrs. under a stream of N gave a low yield of 2-methyl-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline, m. 110-11°. A suspension of 7.6 g. IX in 70 cc. dry benzene and 70 cc. Ac2O refluxed (water bath) 20 min. gave 6.6 g. 3-carboxy-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-2-acetic acid cyclic anhydride (X), m. 215-16° (decomposition). A mixture of 40 mg. X, 23 mg. 3,4-dimethoxyphenethylamine, and 5 cc. CHCl3 refluxed 1.5 hrs. gave 50 mg. of product, m. 142-3° (decomposition). Spectral data were compatible with the structure, 3,4-dimethoxyphenethylammonium 8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-2-(3,4-dimethoxyphenethyl)acetamide-3-carboxylate. A solution of 6 g. X in 120 cc. 1:1 pyridine-EtOH refluxed 15 min. gave, after standing overnight and crystallizing from ether, 5.5 g. Et 3-carboxy-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-2-acetate (XI), m. 148° (decomposition). A solution of 2.5 g. XI in 25 cc. CHCl3 was treated with 2 successive portions of 1 g. dicyclohexylcarbodiimide (allowing to stand 24 hrs.) and 0.62 g. 3,4-dimethoxyphenethylamine (allowing to stand 15 addnl. hrs.) to yield 2.4 g. Et 3-[(3,4-dimethoxyphenethyl)carbamoyl]-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-2-acetate (XII), m. 132'. A solution of 1 g. XII in 30 cc. toluene refluxed several hrs. with 5 g. P2O5 gave 455 mg. Et 3-(6,7-dimethoxy-3,4-dihydro-1-isoquinolyl)-8,9-dimethoxy-5,6-dihydropyrrolo [2,1-a]isoquinoline-2-acetate (XIII), m. 136°. Reduction of 400 mg. XIII in a mixture of 20 cc. absolute tetrahydrofuran and 30 cc. ether with 59.7 mg. LiAlH4 yielded 243 mg. 3-(6,7-dimethoxy-3,4-dihydro-1-isoquinolyl)-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-2-ethanol (XIV), m. 163-4°. A solution of 105 mg. p-toluenesulfonyl chloride in 5 cc. absolute benzene was added to a solution of 255 mg. XIV in 20 cc. absolute benzene. After standing overnight there was deposited 329 mg. bisnorrubremetinium p-toluenesulfonate (Ia), orange-yellow, m. 192-4° (H2O) (after drying 15 hrs. in vacuo over P2O5 at 95°). KBr (58 mg.) in 5 cc. H2O was added to a solution of 200 mg. Ia in 30 cc. H2O and the mixture warmed. On cooling there was deposited 160 mg. bisnorrubremetinium bromide (Ib). Recrystn. from 4% KBr solution and H2O gave orange-yellow needles, m. 215°, after drying similarly. The irspectrum was identical with that of Ib from another source. 32 references.

AN 1965:463358 CAPLUS
DN 63:63358
OREF 63:11644a-h,11645a-c
TI Structure of (+)-rubremetinium cation. New synthesis of C-bisnorrubremetinium cation and its model compound
AU Ban, Yoshio; Terashima, Masanao
CS Hokkaido Univ., Sapporo, Japan
SO Chemical & Pharmaceutical Bulletin (1965), 13(7), 775-85
CODEN: CPBTAL; ISSN: 0009-2363
DT Journal
LA English
OS CASREACT 63:63358
IT 3381-96-2
RL: PREP (Preparation)
(Derived from data in the 7th Collective Formula Index (1962-1966))
RN 3381-96-2 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-3-carboxamide, N-[2-(3,4-dimethoxyphenyl)ethyl]-5,6-dihydro-8,9-dimethoxy-2-methyl- (CA

INDEX NAME)



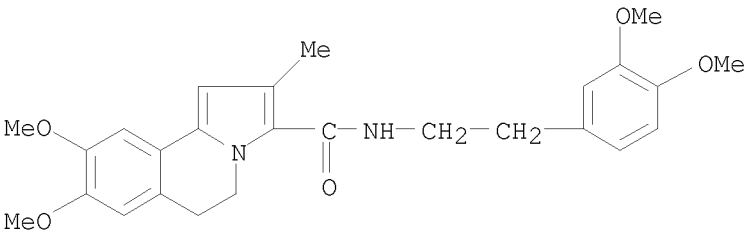
L7 ANSWER 87 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB cf. CA 63, 5697c. The bark of the species A. dasycarpon was found to contain 12 alkaloids. The total MeOH extract (800 g.) in 600 ml. AcOH stirred into 4 l. H2O 6 hrs. and the filtered solution washed with petr. ether and extracted with 1500 ml. C6H6 gave 1.9 g. fraction A. The pH adjusted to 7.0 and the solution extracted with 1500 ml. CHCl3 yielded 6.1 g. fraction B. Extraction with 1500 ml. CHCl3 after readjustment of the pH to 11.0 produced 3.4 g. fraction C. Fraction A chromatographed from Et2O over Al2O3 gave the known crystalline uleine (I, R = CH2, R' = Me) (II), m. 75-98° (MeOH), [α]25D 16.5° (c 0.91, all in CHCl3).
Fraction B chromatographed over Al2O3 gave 10 cuts from C6H6 to Et2O and from all fractions II (8.3 g.) crystallized separately. Filtrates from the 1st 5 fractions evaporated and the residues crystallized from Me2CO gave 400 mg. (+)-apparicine (III), m. 192-4°, [α]27D 176.0° (c 2.16). Thin-layer chromatography (TLC) of the 2nd fraction [on silica gel from 4:4:2 EtOAc-C6H6-EtOH, sprayed with 2% Ce2(SO4)3 in M H2SO4 or irradiated with uv light] isolated 20 mg. (zone at Rf 0.5) Nb-methyltetrahydroellipticine (IV), m. 198-200° (Me2CO). TLC of the 3rd fraction isolated 100 mg. dasycarpidone I (R = O, R' = MeO) (V), [α]26D 64.7° (c 1.02). TLC of fraction 7 and crystallization of zone Rf 0.65 from Et2O gave 2 mg. polyneuridine (or akuammidine) aldehyde (VI), m. 231-3°. Crystallization of zone Rf 0.5 gave (+)-guatambuine (VII), m. 236-8° (decomposition), [α]25D 88° (c 0.67, dioxane). Zone Rf 0.4 crystallized from CHCl3 yielded 30 mg. des-N-methyldasycarpidone (I, R = O, R' = H) (VIII), m. 208-10°. Concentration of fraction 8 in vacuo gave 3 mg. crystalline dehydrodes-N-methyluleine (IX), m. 220°. Fraction C chromatographed over Al2O3 with CHCl3 up to 9:1 CHCl3-MeOH was separated into 5 cuts. Rechromatography of cut 3 in 98:2 CHCl3-MeOH over Al2O3 into 60 fractions and TLC of the residues from fractions 15-53 from 80:15:5 C6H6-Me2CO-Et2NH yielded 35 mg. 1,13-dihydro-13-hydroxyuleine I (R = H,CH2OH, R' = Me) (X), [α]27D -96° (c 0.25, alc.).
Rechromatography of cut 4 from 98:2 CHCl3-MeOH over Al2O3 gave (from fractions 12-15) 125 mg. des-N-methyluleine I (R = CH2, R' = H) (XI), m. 143-5° (CHCl3), [α]26D -20° (c 1.18, alc.). Fractions 26-32 gave 210 mg. dasycarpidol (I, R = H, OH, R' = Me) (XII), m. 118-22° (CHCl3), [α]26D -54° (c 1.03, alc.). Cut 5 rechromatographed from 99:1 CHCl3-MeOH yielded, from fractions 9-21, 150 mg. aspidodasycarpine (XIII, R = CH2OH, R' = H) (XIV), m. 207-9° (Me2CO), [α]25D -101° (c 1.42). II, IV, and VII were isolated and studied previously and were 3 of the 8 known complex indole alkaloids lacking the tryptamine 2 carbon bridge. It was of considerable

interest and biogenetic significance to isolate 7 new indolic compds. (III, V, VIII, IX, X, XI, XII) all lacking this feature. Both the remaining alkaloids (VI, XIV) have the usual 2 carbon functions at the indolic β position. Alkaloids III, V, VIII, X, XI, and XII were the subject of previous publications. XI (30 mg.) treated with 0.5 ml. MeI in 6 ml. refluxing 1:1 Me2CO-C6H6 30 min. gave 10 mg. II MeI salt, m. 196-8°. The evaporated filtrate treated with aqueous NaOH and extracted with CHCl3 gave 11 mg. II, m. 70-5° (MeOH), [α]25D 6.7° (c 0.6). Treatment of XI with Ac2OC5H5N 16 hrs. at 20° and partition of the residue on evaporation with Et2O and aqueous K2CO3 gave des-N-methyl-N-acetyluleine I (R = CH2, R' = Ac) (XV), m. 214-15°. XV reduced with excess LiAlH4 in refluxing tetrahydrofuran 16 hrs. and the isolated product purified by preparative TLC gave 4 mg. colorless glassy N-ethyldes-N-methyluleine I (R = CH2, R' = Et) (XVI), m/e 280 (93%). XVI treated with MeI in C6H6 30 min. at 20° gave des-N-methyl-N-ethyluleine methiodide (XVII), m. 190-1°, mixed m.p. with uleine ethiodide (XVIII), m. 198-201°. VIII (7 mg.) in 2 ml. 1:1 C6H6-Me2CO refluxed 1 hr. with 0.3 ml. MeI and the residue on evaporation treated with 0.1N NaOH and Et2O yielded 2 mg. V. Chemical verification for the structural assignment for V was provided by conversion of II to V. Low temperature ozonolysis of 350 mg. II in 21 ml. 20:1 EtOAc-MeOH, stirring with 3 g. Zn in 5 ml. AcOH and chromatography of the isolated product gave 47 g. V, [α]27D 61.2° (c 0.91), also produced by CrO3-C5H5N oxidation of XII. Analysis of the N.M.R. spectrum of XII and inspection of Dreiding models led to an expression for the relative stereochemistry of the mol. The partial synthesis of X from II confirmed the identity of their skeleton structures. The addition of the elements of H2O to II in the desired orientation was achieved by hydroboration. II treated with BF3.Et2O in Et2O and the salt (170 mg., m. 205°) in 3 ml. tetrahydrofuran stirred 16 hrs. with 210 mg. NaBH4 and 0.2 ml. BF3.Et2O, the mixture refluxed 5 hrs. with 1 ml. 30% NaOH and 2 ml. 30% H2O2 and the product chromatographed on Al2O3, eluted with 99:1 CHCl3-MeOH and the eluate evaporated gave 115 mg. X, [α]26D -97° (c 0.7, alc.), identical with that of the naturally occurring alc. showing that the absolute configuration of X is the same as that of II. XIV acetylated with 1:1 Ac2O-C5H5N 16 hrs. at 20° gave aspidodasycarpine N,O-diacetate (XIII, R = CH2OAc, R' = Ac) (XIX), m. 111-14°, [α]29D -34.5° (c 1.42). XIX was non-basic and unreactive with MeI at 20°. These facts and the unchanged uv absorption necessitated that the basic Nb be secondary and that the 3rd O atom be alcoholic, with the remaining O ethereal. Base catalyzed retroaldolization of XIX and purification of the gummy product by preparative TLC (band at Rf 0.7) gave amorphous XIII (R = H, R' = Ac) (XX), [α]25D -86° (c 0.98, alc.). Information on the nature of the ring containing Nb and definite evidence as to the ethereal nature of the 4th O atom and its site of attachment was obtained from XIII (R = H, R' = CH2OH) (XXI) and its reduction products. XIV (20 mg.) refluxed with excess MeONa in 5 ml. MeOH 6 hrs. under N and the isolated product chromatographed from CHCl3 over Al2O3 to give 7 mg. XIII (R = R' = H) (XXII), acetylated with Ac2O-C5H5N to give XX. XIV (40 mg.) in 2 ml. MeOH kept 30 hrs. at 20° with 100 mg. KOH in 1 ml. H2O and filtered gave 27 mg. crystalline XXI, m. 175-82° (decomposition), [α]30D -50° (c 0.18), also prepared by treating XXII in 2:1 MeOH-H2O 1 hr. at 20° with KOH and HCHO. XXI was easily reconverted to XXII by treatment with AcOH. Reduction of XXI with LiAlH4 in tetrahydrofuran 8 hrs. under reflux and purification of the isolated product by preparative TLC (zone at Rf 0.1) gave gummy XXIII

(R = H, R1 = Me, R2 = H, R3 = CH2OH) (XXIV). Similar reduction using LiAlD4 gave the corresponding deuterio derivative XXIII (R = D, R1 = CDH2, R2 = H, R3 = CD2OH) (XXV). With the establishment of a carbinolamine ether system at Na it became clear why this N atom was not acetylated during the treatment of the alkaloid with Ac2O-C5H5N. XIV (10 mg.) in 3 ml. 1:2 Me2CO-C6H6 treated with MeI at 20°, the Et2O-washed precipitate treated with 0.1N NaOH and extracted with CHCl3 gave 4 mg. N-methylaspidodasycarpine XIII (R = CH2OH, R' = Me) (XXVI). LiAlH4 reduction of XIX gave amorphous XXIII (R = H, R1 = Et, R2 = R3 = CH2OH) (XXVII). Similar reduction of XX with LiAlD4 gave the alc. XXIII (R = D, R1 = CD2Me, R2 = H, R3 = CD2OH) (XXVIII). The mass spectral base peaks of XXVII and XXVIII are consistent with the assumption that the basic N atom forms part of a piperidine ring with a 2 carbon substituent (ethylidene group), and thus permitting expansion of the partial structure for XIV. Treatment of XIX with Zn and HCl gave, in addition to the anticipated dihydroindolinic compound XXIII (R = H, R1 = Ac, R2 = MeO2CCH2, R3 = CO2Me) (XXIX), the indole secoaspidodasycarpine-N-acetate (XXX), m. 196.9° (C5H5N-H2O). Acetylation of XXX gave the N,O-diacetate (XXXI), m. 187-90° (MeOH-H2O). Analysis of the N.M.R. spectrum of XXX suggested that the oxygenated side chain must represent the other end of the ether linkage already established as being attached to C-2 in the expanded partial structure. Strong indications as to the sites of attachment of the indole system and the unsatd. ester moiety to the piperidine nucleus were gained from the N.M.R. spectrum of XXX, and from peaks in the mass spectrum at m/e 194 and 236. The structure of these ions were confirmed by 4-mass unit shifts to m/e 198, 240 by reduction of XXX in alc. over 10% Pd-C to the tetrahydro compound On the assumption that no fundamental skeletal rearrangement occurred during the production of XXX the partial structure of XIV was extended to the given structure (XXXII) in which necessarily atoms C-7 and C-16 must be joined to arrive at the given structure XIII (R = CH2OH, R' = H) for XIV. Chemical confirmation for the structure of XIV was obtained by interconversion with picraline (XXXIII) of established structure and absolute configuration. XXXIII (98 mg.) and 2 g. KBH4 in 20 ml. MeOH refluxed 5 hrs. and the cooled solution acidified with 2N HCl, concentrated in vacuo, made alkaline with NaOH, extracted with Et2O, and the solvent evaporated gave a glass with ir spectrum and TLC mobility identical with those of XXII. The glass (50%) in 1.5 ml. 2:1 MeOH-H2O kept 3 hrs. with 80 mg. KOH and 2 drops of 37% aqueous HCHO gave 22 mg. XXI, identical with material obtained from XIV. The ir spectrum and TLC mobility of XX prepared from the NaBH4 reduction product XXV of XXXIII with C5H5N-Ac2O was also identical with XXII derived from XIV. The mechanism of formation of XXX appeared to be a fragmentation reaction and the proposed scheme was consistent with the isolation of secoaspidodasycarpine by Zn-HCl treatment of XIV. The co-occurrence of uleine type alkaloids with a base in which the tryptamine bridge-Nb bond has been broken is of interest in view of the suggestion by Wenkert (CA 57, 1285e) that the uleine type mol. is formed from an indole moiety which does not yet contain such a 2-atom bridge. Cf. CA 63, 10007c.

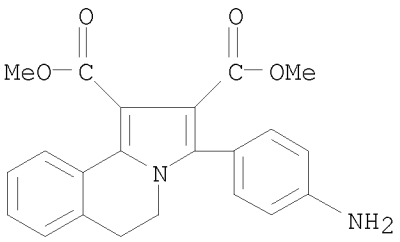
AN 1965:463357 CAPLUS
DN 63:63357
OREF 63:11641h,11642a-h,11643a-h,11644a
TI Alkaloid studies. LIII. Structures of nine new alkaloids from Aspidosperma dasycarpon
AU Joule, J. A.; Ohashi, M.; Gilbert, B.; Djerassi, Carl

CS Stanford Univ., Stanford, CA
SO Tetrahedron (1965), 21(7), 1717-34
CODEN: TETRAB; ISSN: 0040-4020
DT Journal
LA English
IT 3381-96-2
(Derived from data in the 7th Collective Formula Index (1962-1966))
RN 3381-96-2 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-3-carboxamide, N-[2-(3,4-dimethoxyphenyl)ethyl]-5,6-dihydro-8,9-dimethoxy-2-methyl- (CA INDEX NAME)



L7 ANSWER 88 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB 2-Alkyl-3,4-dihydroisoquinolinium salts, obtainable by the alkylation of 3,4-dihydroisoquinoline or from o-BrCH2CH2C6H4CHO with primary amines, treated in hot C5H5N with Et3N yielded solns. of the corresponding azomethinylides I. Thus, 2-(p-nitrobenzyl)-3,4-dihydroisoquinolinium bromide gave an orange solution of I (R = p-O2NC6H4) (II), which added readily in situ to di-Me fumarate (III) to yield 69% IV. IV treated in boiling xylene with chloranil gave V, hydrogenated over Ni to yield VI. The successive deamination, hydrolysis, decarboxylation, and dehydrogenation of VI gave 3-phenylbenzo[g]pyrrocoline, which was also prepared by the dehydrocyclization of 1-(3-phenylpropyl)isoquinoline over Cu chromite at 590°. Cycloadducts similar to IV were also obtained from II with CH2:CHCO2Me (VII), trans-(:CHCN)2 (VIII), CH2:CHCN (IX), (BzCH:)2 (X), and BzC.tplbond.CPh (XI) in 69, 36, 42, 69, and 22% yield, resp. The addition of II to CS2 proceeded with the loss of 2H and the formation of 65% mesoionic XII, copper-red plates. I (R = Bz) (XIII), obtained from 2-phenacyl-3,4-dihydroisoquinolinium bromide with Et3N or C5H5N in MeCN at 20-80°, yielded by addition to XI (with simultaneous dehydrogenation) 45% XIV. XIII added to (.tplbond.CCO2Me)2 and HC.tplbond. CCO2Me in 28% yield each; these cyclo-addns. were also accompanied by the aromatization of the 5-membered hetero ring. XIII yielded 1:1 adducts with VII, III, N-phenylmaleimide, trans-X, IX, and VIII in 50, 73, 73, 76, 55, and 19% yield, resp., and with PhCH:NMe, PhCH:NPh, PhNCO, PhNCS, and CS2 in 40, 54, 66, 72, and 87% yield, resp. The orange betaine from 2-(p-nitrobenzyl)isoquinolinium chloride also can be regarded as an azomethinylide; it added in CHCl3 to PhNCO to yield 55% black-red, tryst. XV.
AN 1963:482198 CAPLUS
DN 59:82198
OREF 59:15255d-h,15256a-b
TI Azomethinylides and their 1,3-dipolar cycloadditions

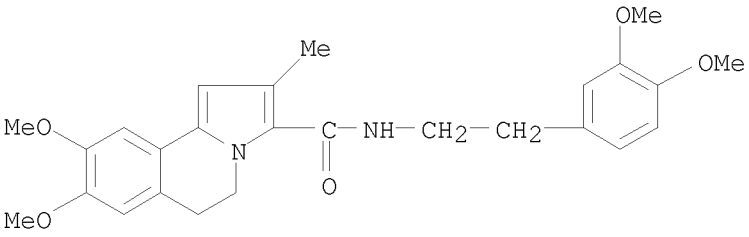
AU Huisgen, Rolf; Grashey, Rudolf; Steingruber, Elmar
CS Univ. Munich, Germany
SO Tetrahedron Letters (1963), (22), 1441-5
CODEN: TELEAY; ISSN: 0040-4039
DT Journal
LA German
OS CASREACT 59:82198
IT 100407-10-1P, Pyrrolo[2,1-a]isoquinoline-1,2-dicarboxylic acid, 3-(p-aminophenyl)-5,6-dihydro-, dimethyl ester
RL: PREP (Preparation)
(preparation of)
RN 100407-10-1 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-1,2-dicarboxylic acid, 3-(4-aminophenyl)-5,6-dihydro-, 1,2-dimethyl ester (CA INDEX NAME)



L7 ANSWER 89 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB The correctness of the Battersby, et al. (CA 43, 1492a), formulation for the rubremetinium cation (I) was confirmed by a synthesis not involving an oxidation process in the final stage. AcCHClCO2Et and 3,4-dihydro-6,7-dimethoxy-1-methylisoquinoline refluxed in alc. yielded 94% ester (II, R = Me, X = OEt), m. 91.0-2.0°; hydrazide m. 197-8°. Hydrolysis of the ester with 10% alc. KOH gave 97% free acid, II (R = Me, X = OH) (III), m. 160-2° (decomposition), readily decarboxylated by heating in C6H6 to give a quant. yield of II (R = Me, COX = H) (IV), m. 110.0-10.5°. III, 3,4-(MeO)2C6H3CH2CH2NH2, and dicyclohexylcarbodiimide in CHCl3 kept at room temperature yielded 23% II [R = Me, X = 3,4-(MeO)2C6H3CH2CH2NH], m. 171-3°, cyclized with P2O5 to yield 46% free base (V, R = Me) (VI), m. 165-7°, converted with Me2SO4 to red prismatic needles of the quaternary base (VII), m. 176-8°. The absorption spectra of VI in 0.01N HCl and of VII were similar but not identical with that of I. The chromophoric systems were not free from the effect induced by the inhibition of free rotation of the C-3, C-1' axis, and accordingly a new synthesis of bisnorrubremetinium salt was successfully attempted, substituting EtO2CCH2COCHClCO2Et for AcCHClCO2Et in the above synthesis. Refluxing the diester with the isoquinoline in alc. yielded 54% II (R = CH2CO2Et, X = OEt), m. 95-7°, hydrolyzed to yield 86.5% dicarboxylic acid, m. 160-1° (decomposition), decarboxylated by heating in xylene to give a poor yield of IV. The acid refluxed 20 min. with Ac2O in C6H6 yielded the corresponding anhydride, m. 216° (decomposition), refluxed in alc. C5H5N to yield 79% half-ester, II (R = CH2CO2Et, X = OH) (VIII), m. 148° (decomposition). VIII, 3,4-(MeO)2C6H3CH2CH2NH2, and dicyclohexylcarbodiimide in CHCl3 at room temperature yielded 66% amide, II [R = CHCO2Et, X =

3,4-(MeO)2C6H3CH2CH2NH], m. 132°, boiled 5 hrs. with P2O5 in PhMe to give 47.2% V (R = CH2CO2Et), m. 136°, reduced by refluxing 3 hrs. at LiAlH4 in Et2O-tetrahydrofuran to the corresponding alc., V (R = CH2CH2OH) (IX), m. 163-4°, with ultraviolet absorption spectrum reasonably identical with those of the ester and VI. IX and p-MeC6H4SO2Cl kept in C6H6 at room temperature gave the tosylate, m. 192-4°, readily converted to the corresponding bromide, m. 215°, with infrared spectrum identical with that of C-bisnorrubremetinium bromide. Since dehydrogenation did not occur in the final step of the synthesis, the formulation I was conformatively supported.

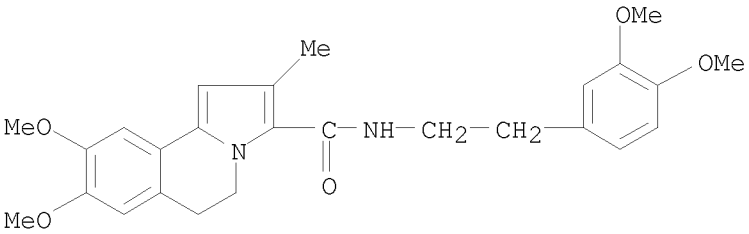
AN 1962:73617 CAPLUS
DN 56:73617
OREF 56:14343f-i,14344a-d
TI Structure of rubremetinium cation. New synthesis of C-bisnorrubrethetinium salt and its model compound
AU Ban, Yoshio; Terashima, Masanao
CS Hokkaido Univ., Sapporo, Japan
SO Tetrahedron Letters (1961) 796-801
CODEN: TELEAY; ISSN: 0040-4039
DT Journal
LA Unavailable
IT 3381-96-2
RL: PREP (Preparation)
(Derived from data in the 7th Collective Formula Index (1962-1966))
RN 3381-96-2 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-3-carboxamide, N-[2-(3,4-dimethoxyphenyl)ethyl]-5,6-dihydro-8,9-dimethoxy-2-methyl- (CA INDEX NAME)



L7 ANSWER 90 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
AB 3-Hydroxy-4-methoxy-2-nitrophenylacetic acid (10 g.) in 10 ml. 1:1 alc.-H2O refluxed 3.5 hrs. with 50 ml. PhCH2Cl, 150 ml. H2O added, and the mixture steam distilled gave a crude benzyl ester, saponified by refluxing 0.5 hr.
with 200 ml. H2O and 160 ml. 5N NaOH to give 11.5 g. 3-benzyloxy-4-methoxy-2-nitrophenyl-acetic acid (I), m. 145-6° (alc.). The acid chloride of I condensed with 3,4-dimethoxyphenylethylamine gave 3'-benzyloxy-4'-methoxy-2'-nitrophenyl-N-[2-(3,4-dimethoxy-phenyl)ethyl]acetamide, m. 108-9°. Recrystn. of the high-melting amide from 80% MeOH gave a product, m. 48-50°. The picrolonate of 1-(2'-amino-3'-benzyloxy-4'-methoxy-benzyl)-N-methyl-6,7-dimethoxytetrahydroisoquinoline was prepared from the formed amide. The picrolonate (1 g.) was suspended in 5 ml. MeOH, and 5 ml. MeOH containing 1

ml. concentrated H2SO4, the MeOH solution of the base cooled, 150 mg. NaNO2 in 10
ml. MeOH added, the mixture left overnight at 5°, 300 mg. catalytic Cu powder added, after 1 hr. the suspension refluxed 0.5 hr., Cu filtered off, the solution extracted with Et2O, the extract discarded, the solution made alkaline,
extracted with Et2O, evaporated, and the 355 mg. of residue chromatographed on Al2O3. Only the fractions containing the products with Rf 0.32 and 0.07 were worked up, as a preliminary hydrolysis with acid showed on paper chromatography that they were related to (±)-isocorydine (II) and (±)-laudanidine (III). Evaporation of the first fractions yielded 13 mg. of crude product; this material refluxed 40 min. with 1 ml. 20% HCl, cooled, extracted with Et2O, made basic, and extracted again with Et2O gave from the exts.
7.5 mg. oily residue. This residue (75 mg.) in C6H6 chromatographed on Al2O3 gave 35 mg. crude II; II.HCl m. 211-12°. II.HCl (30 mg.) in 2 ml. H2O made alkaline and extracted with Et2O gave II, m. 150-2° (MeOH-Et2O). The fractions containing substance with Rf 0.07 united and evaporated gave 130 mg. of an oil. This residue refluxed 40 min. with 10 ml. 20% HCl, extracted with Et2O, made alkaline, and extracted again with Et2O gave 51 mg.
(crude) III, prisms, m. 164-5° (MeOH); picrolonate m. 163-5°.

AN 1962:73616 CAPLUS
DN 56:73616
OREF 56:14343b-f
TI Synthesis of (±)-isocorydine
AU Kuck, A. M.; Frydman, B.
CS E. R. Squibb & Sons Argentina S. A., Martinez
SO Journal of Organic Chemistry (1961), 26, 5253-4
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA Unavailable
IT 3381-96-2
(Derived from data in the 7th Collective Formula Index (1962-1966))
RN 3381-96-2 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-3-carboxamide, N-[2-(3,4-dimethoxyphenyl)ethyl]-5,6-dihydro-8,9-dimethoxy-2-methyl- (CA INDEX NAME)

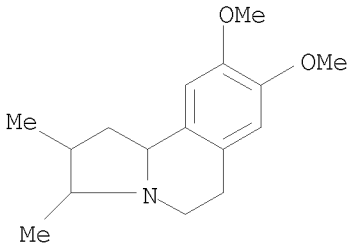


L7 ANSWER 91 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
AB Equivalent amts. of homoveratrylamine and AcCHR(CH2)nCO2Et (R = H and Me, n = 1 and 2), kept 2-5 days in absolute alc. with Pd-C and H at 55-60° (90-100% H absorbed), filtered, the filtrate concentrated at 12 mm., the residue

heated 2 hrs. at 180-90° (12 mm.), and the lactam, (I) which distilled fractionated and redistd. gave 70-80% yields.: 1-(3,4-dimethoxyphenethyl)-5-methyl-2-pyrrolidone, b0.03 140-55°, and 4,5-di-Me analog, b0.2 155-65°; 1-(3,4-dimethylphenethyl)-6-methyl-2-piperidone, b1 170-80°, and the 5,6-di-Me analog, b1 175-85°. I (1 g.) in 10 cc. absolute PhMe, heated 1 hr. with 3 cc. POCl3 (protected from moisture), concentrated in vacuo, taken up in 2 N HCl, the solution washed with ether, made basic with NaOH, extracted with ether (or CH2Cl2), and the extract dried, concentrated, and distilled, gave 70-80% isoquinoline derivs. (II): 8,9-dimethoxy-2,3,5,6-tetrahydro-3-methylbenzo [g] pyrrocoline, b0.02 120-30°, (picrate, m. 154-5°), and the 2,3-di-Me analog, b0.01 135-45° (picrate, m. 171-3°). 9,10-Dimethoxy-3,4,6,7-tetrahydro-4-methyl-2H-benzo[a]quinolizine, b1 160-70° (picrate, m. 153-4°); and the 3,4-di-Me analog, b0.01 135-40° (picrate, m. 137-9°). II kept 2-4 days at room temperature with Pd-C in 50% HOAc and H gave 80-90% dihydro derivs. (III): 8,9-dimethoxy-1,2,3,5,6,10b-hexahydro-3-methylbenzo[g]pyrrocoline, b0.02 110-20° [picrate, m. 173-5°; HCl salt, m. 246-8° (decomposition)]; and 2,3-di-Me analog, b0.02 150-5° [picrate, m. 163-4°; HCl salt, m. 213-16° (decomposition)]. 9,10-di-Methoxy-1,2,3,4,6,7-hexahydro-4-methyl-11bH-benzo[a]quinolizine, b1 150-5° [picrate, m. 181-3°; HCl salt, m. 214-17° (decomposition)]; and 3,4-di-Me analog, b0.01 130-5° [picrate, m. 197-9°; HCl salt, m. 217-19° (decomposition)]. III (1 g.)in 10 cc. absolute C6H6 heated 2 hrs. with 1.7 cc. PhCH2I, kept overnight, the C6H6 decanted, the oily salt washed with C6H6, taken up in 20 cc. 50% MeOH, shaken 2 hrs. with 1.1 g. AgNO3, the solution filtered, concentrated, and the residue heated 1 hr. at 12 mm. on a water bath, taken up in ether, washed with H2O, purified as the HCl salt, and distilled, gave 70-90% degradation product (IV): 1-Benzyl-2-(4,5-dimethoxy-2-vinylphenyl)-5-methylpyrrolidine, b0.001 125-35°, and 4,5-di-Me analog, b0.01 130-40°. 1-Benzyl-2-(4,5-dimethoxy-2-vinylphenyl)-6-methylpiperidine, b0.02 150-60°, and 5,6-di-Me analog, b1 180-90°. IV (1 g.) in 20 cc. 50% HOAc, kept 2-3 hrs. at room temperature with Pd-C and H, then 5-10 hrs. at 55-60°, gave 90-95% bases: 2-(4,5-dimethoxy-2-ethylphenyl)-5-methylpyrrolidine, b0.002 105-15° (picrate, m. 154-6°); and 4,5-di-Me analog, b0.2 125-35° (picrate, m. 166-7°). 2-(4,5-Dimethoxy-2-ethylphenyl)-6-methylpiperidine, b1 140-5° (picrate, m. 205-6°), and 5,6-di-Me analog, b1 130-40° (picrate, m. 197-200°).

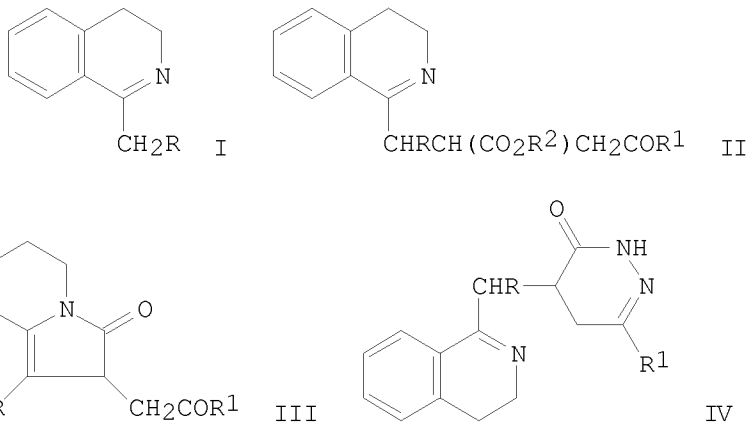
AN 1953:12212 CAPLUS
DN 47:12212
OREF 47:2187f-i,2188a-c
TI Synthesis of compounds with constitutional reference to emetine. III. Synthesis of benzo[g]pyrrocoline and 11bH-benzo[a]quinolizine
AU Pailer, M.; Brandstetter, W.
CS Univ. Vienna
SO Monatshefte fuer Chemie (1952), 83, 523-9
CODEN: MOCMB7; ISSN: 0026-9247
DT Journal
LA Unavailable
IT 853924-96-6, Benzo[g]pyrrocoline, 1,2,3,5,6,10b-hexahydro-8,9-dimethoxy-2,3-dimethyl-
RL: PREP (Preparation)

(and derivs.)
RN 853924-96-6 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline, 1,2,3,5,6,10b-hexahydro-8,9-dimethoxy-2,3-dimethyl- (CA INDEX NAME)



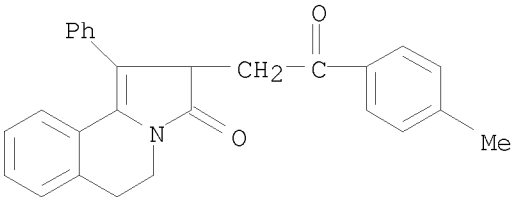
=> d abs bib fhitstr 70-79

L7 ANSWER 70 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI



AB Treatment of I (R = H, Ph) with R1COCH:CHCO2Me (R1 = Ph, p-tolyl, 4-MeOC6H4, tetrahydro-2-naphthyl) gave 43-91% II (R2 = Me). Several II (R2 = H) were prepared similarly in 56-92% yield. II (R2 = H) were esterified with MeOH to give II (R2 = Me). II (R = Ph; R1 = Ph, p-tolyl, tetrahydro-2-naphthyl) in refluxing MePh gave 50-96% III. IV (R = Ph; R1 = p-tolyl; tetrahydro-2-naphthyl) were prepared from II (R2 = H) by treatment with N2H4.
AN 1979:540700 CAPLUS
DN 91:140700
OREF 91:22695a,22698a
TI Reaction of β-aroylacrylic acids with 1-substituted

3,4-dihydroisoquinolines
AU Agbalyan, S. G.; Khachikyan, R. D.
CS Inst. Org. Khim., Yerevan, 375094, USSR
SO Khimiya Geterotsiklicheskikh Soedinenii (1979), (7), 943-5
CODEN: KGSSAQ; ISSN: 0453-8234
DT Journal
LA Russian
OS CASREACT 91:140700
IT 71483-92-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 71483-92-6 CAPLUS
CN Pyrrolo[2,1-a]isoquinolin-3(2H)-one,
5,6-dihydro-2-[2-(4-methylphenyl)-2-oxoethyl]-1-phenyl- (CA INDEX NAME)

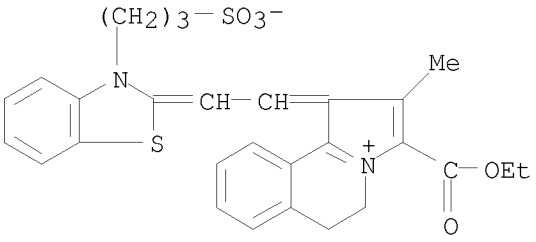


L7 ANSWER 71 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB A direct pos. Ag halide photog. emulsion contains ≥1 spectral sensitizer dye of the general formula I (R = alkyl, cycloalkyl, aralkenyl, aryl, 5-6 membered heterocyclic ring, carboxy, alkoxycarbonyl, carbamoyl, acyl; R1 = H, alkyl, aryl, carboxy, alkoxycarbonyl, carbamoyl, acyl; R2 = alkyl, alkenyl, aralkyl, aryl, substituted alkyl; Z = group of atoms required to complete 5-6 membered N-containing heterocyclic ring, X- = acid anion, m = 0, 1; n = 0,1). The spectral sensitizer is preferably used with a Ag(Br,I) emulsion fogged with a reducing agent and a Au compound, and the emulsion may contain Pinacrptol Yellow as an organic desensitizer. The direct pos. emulsion exhibit good sensitivity towards blue, green, or red light. Thus, a AgNO3 100 g/500 mL-H2O solution and a KBr 70g/150 mL-H2O solution were added in 50 min to a solution (60°) consisting of gelatin 8 g, 1-N KBr solution 5, and H2O 500 mL, the mixture was kept 5 min at 60°, then a gelatin 75 g/300 mL-H2O solution was added to the mixture, and the emulsion was cooled to solidify it. The emulsion was redissolved, the thiourea dioxide 0.4, and HAuCl4 4.0 mg/mol-Ag were added, the emulsion was fogged 90 min at 60°, then the sensitizwr dye II 300 and Pinacryptol Yellow 200 mg/mol-Ag were added to the emulsion, and the emulsion was coated on a cellulose triacetate film support to give a direct pos. film. The film was sensitometrically exposed and developed to give relative sensitivity, γ-value, Dmax and Dmin of 178, 5.1, 3.13, and 0.03, resp., vs. 100, 5.0, 3.20, and 0.03, resp. for a II-free control. The direct pos. film showed good sensitivity in the wavelength range of 480-600 nm.
AN 1978:180233 CAPLUS
DN 88:180233
OREF 88:28210h,28211a
TI Direct positive silver halide photographic emulsion

IN Tanaka, Akira; Nakatani, Mamoru; Yoshida, Akio
PA Mitsubishi Paper Mills, Ltd., Japan
SO Jpn. Kokai Tokkyo Koho, 12 pp.
CODEN: JKXXAF
DT Patent
LA Japanese
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 52120824	A	19771011	JP 1976-36935	19760402
	JP 54034611	B	19791027		
	US 4147554	A	19790403	US 1977-778328	19770316
PRAI	JP 1976-36935	A	19760402		

IT 66337-41-5
RL: TEM (Technical or engineered material use); USES (Uses)
(photog. sensitizer, for direct-pos. emulsions)
RN 66337-41-5 CAPLUS
CN 1H-Pyrrolo[2,1-a]isoquinolinium, 3-(ethoxycarbonyl)-5,6-dihydro-2-methyl-1-[[3-(3-sulfopropyl)-2(3H)-benzothiazolylidene]ethylidene]-, inner salt (9CI) (CA INDEX NAME)



L7 ANSWER 72 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
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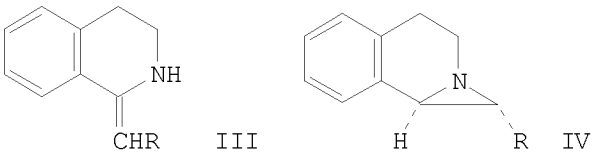
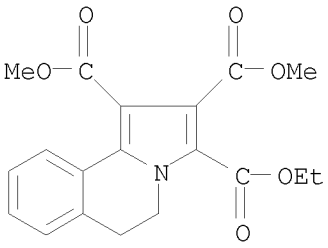
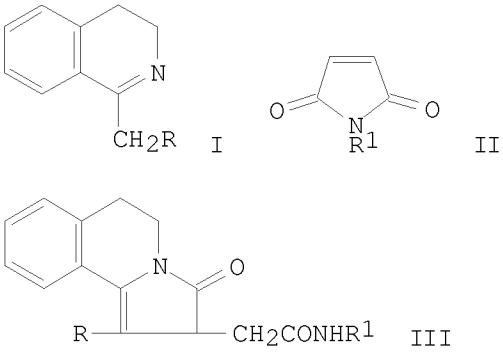


AB 3,4-Dihydroisoquinoline N-oxide (I) reacted with (EtO)2P(O)CH2R (II; R = CN) in MeO(CH2)2OMe-NaH to give 84% enaminonitrile III (R = CN). Similar treatment of I with II (R = CO2Et, CO2Me) gave the enaminonitriles III and the fused aziridines IV (R = CO2Et, CO2Me, resp.). The ratio of III:IV is dependent on the reaction conditions; when using MeO(CH2)2OMe as solvent, IV is the major product, whereas using alc. solvents, the yield of III increases, at the expense of IV, with increasing acidity of the solvent.
AN 1977:601271 CAPLUS
DN 87:201271
OREF 87:31863a,31866a

TI Nitrones. III. Reaction of 3,4-dihydroisoquinoline N-oxide with phosphonoylides
AU Breuer, Eli; Zbaida, Shmuel; Pesso, Joseph; Ronen-Braunstein, Ilana
CS Sch. Pharm., Hebrew Univ., Jerusalem, Israel
SO Tetrahedron (1977), 33(10), 1145-8
CODEN: TETRAB; ISSN: 0040-4020
DT Journal
LA English
OS CASREACT 87:201271
IT 64924-27-2P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and dehydrogenation of)
RN 64924-27-2 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-1,2,3-tricarboxylic acid, 5,6-dihydro-, 3-ethyl 1,2-dimethyl ester (CA INDEX NAME)



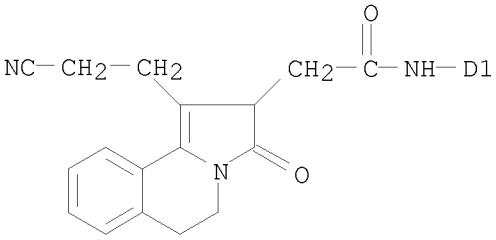
L7 ANSWER 73 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI



AB Reaction of isoquinolines I (R = H, Ph, CO2Me, CH2CH2CN) with maleimides II (R1 = Ph, H, MeOC6H4) gave 25-41% amides III.
AN 1977:5287 CAPLUS
DN 86:5287
OREF 86:911a,914a

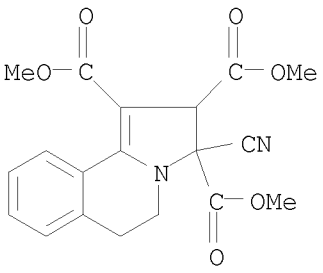
TI Reaction of 1-substituted 3,4-dihydroisoquinolines with maleimide and N-arylmaleimides
AU Agbalyan, S. G.; Khachikyan, R. D.; Lulukyan, K. K.
CS Inst. Org. Khim., Yerevan, USSR
SO Armyanskii Khimicheskii Zhurnal (1976), 29(6), 527-9
CODEN: AYKZAN; ISSN: 0515-9628
DT Journal
LA Russian
OS CASREACT 86:5287
IT 61211-17-4P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 61211-17-4 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-2-acetamide, 1-(2-cyanoethyl)-2,3,5,6-tetrahydro-N-(methoxyphenyl)-3-oxo- (9CI) (CA INDEX NAME)

D1—O—Me

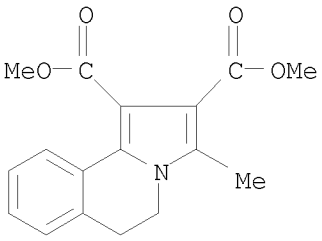


L7 ANSWER 74 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB The isoquinolinium dialkoxycarbonyl ylide I (R = CO2Me, R1 = Me) with R2O2CC.tplbond.CCO2R2, (R2 = Me, Et) in MeOH gave the dihydropyrroloisoquinolines II. The ylides I (R = CO2R1, R1 = Me, Et) in MeOH alone gave the more reactive monoalkoxycarbonyl ylides I (R = H) and R12CO3; the former dimerized to III or reacted in situ with olefins to give 1,2,3,10b-tetrahydropyrrolo[2,1-a]isoquinolines.
AN 1975:593045 CAPLUS
DN 83:193045
OREF 83:30349a,30352a
TI Reactions of isoquinolium ylides. Anomalous products from acetylenes and olefins
AU Basketter, Norman S.; Plunkett, A. Owen
CS Dep. Chem., Portsmouth Polytech., Portsmouth, UK
SO Journal of the Chemical Society, Chemical Communications (1975), (15), 594-5

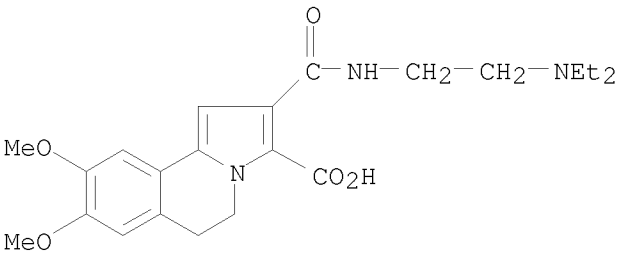
CODEN: JCCCAT; ISSN: 0022-4936
DT Journal
LA English
IT 57699-25-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(demethoxycarbonylation of)
RN 57699-25-9 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-1,2,3-tricarboxylic acid, 3-cyano-2,3,5,6-tetrahydro-, 1,2,3-trimethyl ester (CA INDEX NAME)



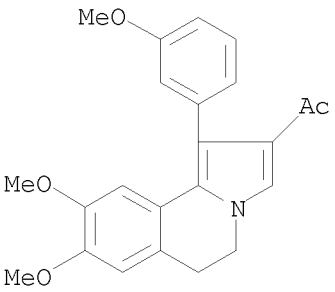
L7 ANSWER 75 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB The reactions of mesoionic oxazolium-5-oxides (munchnones) derived from 1,2,3,4-tetrahydro-1-isoquinolinecarboxylic acids (I, II, III) involve the 1,3-dipolar cycloaddn. to the acetylenic dipolarophiles, MeO2CC.tplbond.CCO2Me and PhC.tplbond.CH. In the latter case, the reaction was regiospecific and gave only IV and V, resp. An isomeric pyrrolo[2,1-a]isoquinoline (VI) was prepared by an unambiguous route and a comparison of the PMR spectra of IV and VI is presented. Photocyclization in MeOH solution of IV in the presence of trace amts. of iodine gave the iodolizinophenanthrene VII. Unsuccessful attempts at the preparation of the analog, VIII, via photocyclization or Pschorr cyclization reactions is also discussed.
AN 1975:139923 CAPLUS
DN 82:139923
OREF 82:22351a,22354a
TI Synthesis of ring-fused pyrroles. II. 1,3-Dipolar cycloaddition reactions of muchnone derivatives obtained from tetrahydroisoquinoline-1-carboxylic acids
AU Hershenson, Fred M.
CS Dep. Chem. Res., Searle Lab., Chicago, IL, USA
SO Journal of Organic Chemistry (1975), 40(6), 740-3
CODEN: JOCEAH; ISSN: 0022-3263
DT Journal
LA English
OS CASREACT 82:139923
IT 53927-34-7P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 53927-34-7 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-1,2-dicarboxylic acid, 5,6-dihydro-3-methyl-, 1,2-dimethyl ester (CA INDEX NAME)



L7 ANSWER 76 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Several monoamides of dihydropyrrolo[2,1-a]isoquinoline-2,3-dicarboxylic acid (I; R = NH2, H2NCH2CH2NH, Et2NCH2CH2NH, morpholinoethylamino, etc.; R1 = Me, R2 = Me, H, PhCH2, R1R2 = CH2) were prepared from the anhydride (II). Several reactions of I were used to determine the structure. II (R1 = R2 = Me) and Et2NCH2CH2OH gave I (R = Et2NCH2CH2O, R1 = R2 = Me). I-monoamide were infused (i.v.) at 0.5 mg/hr/min to anesthetized dogs and the arterial pressure, left ventricular pressure, maximum rate of rise in left ventricular pressure, heart rate and respiratory rate was measured and related to theophylline. The inotropic effect on cat papillary muscle was determined
AN 1973:71881 CAPLUS
DN 78:71881
OREF 78:11421a,11424a
TI Pyrrolo[2,1-a]isoquinoline derivatives. II. Monoamides of 5,6-dihydropyrrolo[2,1-a]isoquinoline-2,3-dicarboxylic acids
AU Casagrande, C.; Invernizzi, A.; Ferrini, R.; Miragoli, G.
CS Res. Lab., Simes S.p.A., Milan, Italy
SO Farmaco, Edizione Scientifica (1972), 27(12), 1029-44
CODEN: FRPSAX; ISSN: 0430-0920
DT Journal
LA English
IT 17606-23-4
RL: RCT (Reactant); RACT (Reactant or reagent)
(N-(dimethylaminoethyl)-8,9-dimethoxy-5,6-dihydropyrrolo[2,1-a]isoquinoline-2,3-dicarboximide from)
RN 17606-23-4 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-3-carboxylic acid, 2-[[[2-(diethylamino)ethyl]amino]carbonyl]-5,6-dihydro-8,9-dimethoxy- (CA INDEX NAME)

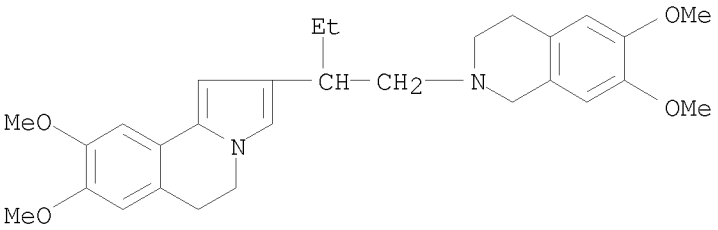


L7 ANSWER 77 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB The title compds. (I) were prepared in quant. yields by cyclization of aroyldihydroiso-quinolines with R1COCH:CHR2. Six I (R = substituted phenyl; R1 = Me, Et; R2 = H, Me) were prepared
AN 1972:501358 CAPLUS
DN 77:101358
OREF 77:16703a,16706a
TI Heterocyclizations of β-iminoketones. Synthesis of 5,6-dihydropyrrolo[2,1-a]isoquinolines
AU Cauwel, Philippe; Gardent, Jean
CS Pharm. Cent., Hop. Paris, Paris, Fr.
SO Tetrahedron Letters (1972), (27), 2781-4
CODEN: TELEAY; ISSN: 0040-4039
DT Journal
LA French
IT 38470-16-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 38470-16-5 CAPLUS
CN Ethanone, 1-[5,6-dihydro-8,9-dimethoxy-1-(3-methoxyphenyl)pyrrolo[2,1-a]isoquinolin-2-yl]- (CA INDEX NAME)



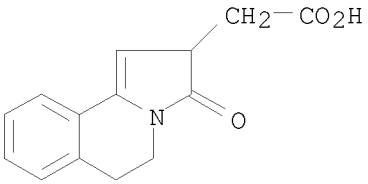
L7 ANSWER 78 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB I was hydrogenated to give, besides the known II and III, IV identified by thin layer chromatog. and ir, uv, and NMR spectra. The yields depended on the reduction method used. Reduction of I with Zn dust-HOAc-H2SO4 gave 50% II and
5% IV, with LiAlH4 35% II and III each, with subsequent hydrogenation over Pt in HOAc for <15 hr .apprx.9% IV, with H/Pt in EtOH for <15 hr or >150 hr 5% II and 10% III or 37.5% II, less III (.apprx.2:1 ratio), and .apprx.1% IV, resp. Hydrogenation of II for 160 hr or of III for 15 hr gave little IV or 30% IV, resp.
AN 1970:477093 CAPLUS
DN 73:77093
OREF 73:12611a,12614a
TI Hydrogenation of rubremetinium salts
AU Kovar, Karl A.
CS Pharm.-Chem. Inst., Univ. Tuebingen, Tuebingen, Fed. Rep. Ger.
SO Archiv der Pharmazie und Berichte der Deutschen Pharmazeutischen

Gesellschaft (1970), 303(7), 579-85
CODEN: APBDAJ; ISSN: 0376-0367
DT Journal
LA German
IT 28674-63-7P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 28674-63-7 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline, 2-[1-[(3,4-dihydro-6,7-dimethoxy-2(1H)-isoquinolinyl)methyl]propyl]-5,6-dihydro-8,9-dimethoxy- (CA INDEX NAME)



L7 ANSWER 79 OF 91 CAPLUS COPYRIGHT 2009 ACS on STN
GI For diagram(s), see printed CA Issue.
AB A mixture of 0.02 mole 1-methyl-3,4-dihydroisoquinoline (I) and 0.02 mole maleic acid was heated to 100° to give 4.9 g. maleate of 1-methyl-3,4-dihydroisoquinoline (II), m. 79-82°. Similarly was prepared 3.8 g. fumarate of 1-methyl-3,4-dihydroisoquinoline (III), m. 127-9°. Heating 4.9 g. II or 3.8 g. III 30 min. at 180° and dissolving the reaction product in a base, gave, resp., after extraction with Et2O and filtration 1.3 and 1.1 g. IV (R = H, R1 = CO2H), m. 190-5° (decomposition). The same acid resulted when 0.02 mole I was heated with 0.02 mole dimethyl maleate or dimethyl fumarate (V). Heating 0.02 mole 1-benzyl-3,4,-dihydroisoquinoline (VI) and 0.02 mole V on a metal bath 3 hrs. at 150-5° gave 2.67 g. IV (R = Ph, R1 = CO2Me), m. 95-7°. Similarly, from diethyl maleate was prepared IV (R = Ph, R1 = CO2Et), m. 80-5°. Heating 0.01 mole 1-substituted 3,4-dihydroisoquinoline and 0.01 mole fumaric acid 2 hrs. at 130-50° gave the following IV (R,R1, yield in g., and m.p. given): (CH2)2CO2Me, CO2H, 1.5, 95-100° (heptane); (CH2)2CN, CO2H, 0.4, 35-8°; CO2Me, CO2H, 1.35, 134-7° (heptane). Heating 0.025 mole I and 0.025 mole methyl cinnamate (VII) 3 hrs. at 160° or 0.1 mole I and 0.1 mole cinnamic acid (VIII) 4 hrs. at 145-50° gave IV (R = H, R1 = Ph), b4 216-20°. Heating 0.04 mole VI with 0.04 mole VIII 15 hrs. at 150-60° or with 0.04 mole VII 14 hrs. at 180-90° gave 7.5 and 9.4 g., resp., IV (R = R1 = Ph), b3 225-30°. Heating 0.03 mole methyl 3,4-dihydroisoquinoline-1-acetate (IX) and 0.03 mole VII 15 hrs. at 160° gave 1.5 g. IV (R = CO2Et, R1 = Ph), b6 240-5°. Similarly, IX and VIII gave 2 g. methyl β-phenyl-α-[1-(3,4-dihydroisoquinolinyl] butyrate, b2 232-4°.
AN 1969:512784 CAPLUS
DN 71:112784
OREF 71:20983a,20986a

TI Activity of the methyl group of 1-methyl-3,4-dihydroisoquinoline. VI. Reactions of 1-substituted 3,4-dihydroisoquinolines with maleic, fumaric, and cinnamic acids and their esters
AU Agbalyan, S. G.; Nersesyan, L. A.
CS Inst. Org. Khim., Erevan, USSR
SO Armyanskii Khimicheskii Zhurnal (1969), 22(8), 714-19
CODEN: AYKZAN; ISSN: 0515-9628
DT Journal
LA Russian
IT 18121-49-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 18121-49-8 CAPLUS
CN Pyrrolo[2,1-a]isoquinoline-2-acetic acid, 2,3,5,6-tetrahydro-3-oxo- (CA INDEX NAME)



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